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METHOD OF SYNTHESIZING A FIRE RETARDANT FOR WOOD

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METHOD OF SYNTHESIZING A FIRE RETARDANT FOR WOOD

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Claims

1. A method of synthesizing a fire retardant for use in the treatment of wood or other fibrous materials in which dicyandiamide, phosphoric acid, boric acid and water are used as the raw materials, characterized by the fact that a reaction of dicyandiamide, phosphoric acid and water is carried out at a temperature of 90-120°C to synthesize dicyandiamidine phosphate with a high yield, and the dicyandiamidine phosphate is then compounded with boric acid to prepare a fire retardant.

2. A method of synthesizing a fire retardant in accordance with Claim 1, characterized by the fact that the mole ratio of dicyandiamidine phosphate to boric acid is 1 : (0.3 to 2.2).

3. A method of synthesizing a fire retardant in accordance with Claim 1, characterized by the fact that the mole ratio of dicyandiamide, phosphoric acid and water is dicyandiamide:phosphoric acid:water = 1:(1.0 to 1.2):(6 to 200).

4. A method of synthesizing a fire retardant in accordance with Claim 1, characterized by the fact that, at the end of the reaction to synthesize dicyandiamidine phosphate, the reaction mixture can be separated by means of crystallization and filtration and the filtration solution can be recovered and recycled.

The present invention pertains to a method of synthesizing a fire retardant.

The conventional method of rendering fire-retarding wood is to treat wood with diammonium hydrogen phosphate and ammonium primary phosphate and other water-soluble inorganic salts. In recent years, new study results are being published all the time (WO 96 00,763, CN 1,093,393). This type of fire retardant has the advantages of having good fire-inhibiting property, low toxicity and low cost of production. However, it also has the serious drawbacks of being easily leached by water, the treated materials have a high moisture absorbency and are prone to condensation and they can corrode metal devices, etc. As a result, their application is restricted. In order to improve the leaching resistance and to reduce the moisture absorbency, many researchers have carried out studies on the use of the phosphate of a low-polymerization amino group compound and an aldehyde-type condensation and polymerization product as a fire retardant for wood. However, despite the fact that the leaching resistance and the moisture absorbency have been significantly improved, the strength of the wood is reduced because a relatively high solidification temperature is needed.

It has been found in recent years that a cooperative fire-retarding effect can be seen not only between phosphorous and nitrogen elements but also among phosphorous, nitrogen and boron elements. Using this cooperative fire-retarding effect and by designing a suitable fire-retarding system, the fire-retarding property can be improved while the amount of the chemical used can be decreased. A fire retardant comprised of the products of an incomplete reaction of dicyandiamide, phosphoric acid, boric acid and water in a temperature of 70-90°C was invented by William J. Oberley (US 4,373,010), which has good fire retarding property and its moisture absorbency is significantly lower than that of an ammonium salt-type fire retardant. However, in order to prevent or to reduce the production of insoluble substances, it is required in said invention that the dicyandiamide and phosphoric acid in the reaction raw materials be converted incompletely and that a balance of 5-10% be present. In this way, because the degree of reaction is difficult to control precisely, the composition of each batch of product is different and the quality of the products is unstable, on the one hand, and on the other hand, there is the drawback that the raw materials cannot be used thoroughly and effectively. In addition, due to the fact that the phosphoric acid is not thoroughly reacted, the acidity of the product is rather high, which causes acid catalyst degradation of the treated wood during the drying process and the quality deteriorates.

The objective of the present invention is to overcome the disadvantages and drawbacks of the prior art method and to provide a method of synthesizing a highly effectively fire retardant with low toxicity suitable for wood and other fibrous materials.

According to the basic concept of the present invention, a thorough reaction of dicyandiamide, phosphoric acid and water (water is used as reaction product and solvent) is carried out at a temperature of 90-120°C to synthesize dicyandiamidine phosphate; the reaction product solution is separated by crystallization and filtration, or by concentration under reduced pressure, drying and other separation methods, to produce a solid dicyandiamidine phosphate, the filtration solution or condensation water can be recovered and recycled; the dicyandiamidine phosphate is mixed with boric acid until uniform and a fire retardant product is thus obtained. The mole ratio of the materials is dicyandiamide:phosphoric acid:water = 1:(1.0-1.2):(6-10); dicyandiamidine phosphate:boric acid = 1:(0.35-2.2).

When the solid fire retardant synthesized by the present invention is dissolved in water to prepare a solution of a suitable concentration, it can be used as a treatment solution for wood or other fibrous materials. Based on the characteristics of the material processing techniques, the fire retardant synthesized by the present invention can also be mixed into a gel material or as a powder additive to be used in the processing of wood artificial board or other fibrous materials.

In the case that no long-term storage or long-distance transportation is involved, a fire retardant product can also be conveniently synthesized directly in liquid form. In other words, the dicyandiamidine phosphate solution obtained at the conclusion of the reaction does not have to go through separation but can be directly added to boric acid, dissolved, and diluted to a suitable concentration to be used later.

The present invention solves the problem that insoluble substances are easily produced in the aqueous solution of a reaction between dicyandiamide and phosphoric acid so that the raw materials can be thoroughly converted to obtain dicyandiamidine phosphate with a high yield and stable quality. It ensures the stability and reliability of the fire retardant product and there is no pollutant discharged from the whole technical process.

Due to the fact that boron compounds are antibacterial and can kill termites, the fire retardant synthesized by the present invention not only has a high fire retardancy but can also impart certain decay-resistant and termite-resistant properties to the treated materials. It is also very low in toxicity and safe to use.

The present invention will be explained in the following with reference to three application examples.

Application Example 1

To a 250 mL four-necked, round-bottomed flask equipped with a temperature gauge, a stirring device and reflux condensation tube and a dropping funnel, 42.1 g dicyandiamide and 80 mL water are added, heating is carried out to 100°C, then, under stirring, 57.7 g of 85% phosphoric acid are added dropwise via the dropping funnel, a reaction is carried out at a temperature of 100-120°C until the dicyandiamide is completely converted. Determination of the end point of the reaction can be based on the change in the pH or by means of the infrared spectrometry. At the end of the reaction, cooling, crystallization and filtration are carried out, followed by washing with 10 mL cold water, then drying is carried out at a temperature of 80-100°C. 74 g of a white dicyandiamidine phosphate product in powder form are obtained with a mole yield of 74%.

1 mol dicyandiamidine phosphate and 1.2 mol boric acid are mixed until uniform to obtain a solid fire retardant product.

To prepare a wood treatment solution, the solid fire retardant synthesized by the present invention is dissolved in water to prepare a clear, colorless and transparent solution with a concentration of 8% (by weight); the pH is about 4.5.

As can be seen from the infrared spectrogram of the synthesized dicyandiamidine phosphate product, there are no absorption peaks characteristic of the cyano group (-CN) at wavelengths 2100 and 2200 (the raw material dicyandiamide has strong absorption at these wavelengths), which indicates that the product does not contain dicyandiamide. The synthesized dicyandiamidine phosphate product is reacted with an excess of lime water such that the phosphoric acid root is completely precipitated in the form of calcium salt. Filtration is carried out, the filtration solution is neutralized with diluted sulfuric acid such that the dicyandiamidine is converted to a sulfate. Concentration, crystallization, separation and drying are then carried out. The infrared spectrogram of the synthesized dicyandiamidine phosphate product conforms completely to the standard Sadtler spectrogram of the standard sample, which indicates that the structure of the product is correct.

Application Example 2

With the exception that the filtration solution produced by the dicyandiamidine phosphate synthesized in Application Example 1 is used to replace the 80 mL water, the dicyandiamidine phosphate synthesis experiment described in Application Example 1 is repeated and 92.0 g of a dicyandiamidine phosphate product is obtained with a mole yield of 92.0%. Experiments are then repeated six times, with the filtration solution of the previous experiment used to replace the water each time, and the mole yield of the dicyandiamidine phosphate is, respectively, 99.5%, 95.5%, 100.2%, 97.8%, 99.0% and 98.0%.

The infrared spectrograms of the dicyandiamidine phosphate products from the above seven repeated experiments conform to the spectrogram of the product of Application Example 1. When 0.5 g of the dicyandiamidine phosphate samples obtained from each of the experiments is respectively dissolved in 4.5 g of distilled water, a clear, colorless and transparent solution with a concentration of 10% is obtained. As is clearly seen, the filtration solution can be recycled for use.

Application Example 3

To a 1000 mL four-necked, round-bottomed flask equipped with a temperature gauge, a stirring device and reflux condensation tube and a dropping funnel, 42.04 g of dicyandiamide and 200 mL water are added, heating is carried out to 100°C, then under stirring, 57.7 g of 85% phosphoric acid are added dropwise via the dropping funnel, a reaction is carried out at a temperature of 100-120°C until the dicyandiamide is completely converted to dicyandiamidine phosphate and heating is stopped. 30.9 g of boric acid and 533 mL of water are added, dissolved, cooled to room temperature, and a liquid fire retardant with a concentration of 15% (by weight) is obtained with a pH of 3.0. To use, it can be diluted with water to a suitable concentration, depending on the need, to prepare a treatment solution for wood.